

Analysis of Particulate and Fiber Debris Samples Returned from the International Space Station

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During the period of International Space Station (ISS) Increments 30 and 31, crewmember reports cited differences in the cabin environment relating to particulate matter and fiber debris compared to earlier experience as well as allergic responses to the cabin environment. It was hypothesized that a change in the cabin atmosphere's suspended particulate matter load may be responsible for the reported situation. Samples were collected and returned to ground-based laboratories for assessment. Assessments included physical classification, optical microscopy and photographic analysis, and scanning electron microscopy (SEM) evaluation using energy dispersive X-ray spectrometry (EDS) methods. Particular points of interest for assessing the samples were for the presence of allergens, carbon dioxide removal assembly (CDRA) zeolite dust, and FGB panel fibers. The results from the physical classification, optical microscopy and photographic analysis, and SEM EDS analysis are presented and discussed.

Nomenclature

<i>cm</i>	=	centimeter
<i>GMT</i>	=	Greenwich Mean Time
<i>UT</i>	=	Universal Time
<i>wt%</i>	=	weight percent
μm	=	micrometer

I. Introduction

DURING a period spanning segments of International Space Station (ISS) Increments 30 and 31 (December 2011-July 2012), crewmember reports cited differences in the cabin environment compared to earlier experience as well as allergic responses to the cabin environment. It was hypothesized that a change in the cabin atmosphere's suspended particulate matter load may be responsible for the reported situation. Concerns were also expressed with respect to Carbon Dioxide Removal Assembly (CDRA) zeolite pellet dust containment and Russian Functional Cargo Block (FGB) panel fabric deterioration potentially adding to the atmospheric particulate and fiber load. As a result of these crewmember reports coupled with the concern over CDRA zeolite pellet dust and FGB panel material deterioration, particulate and debris samples were collected using Kapton[®] tape and returned in late April 2012 aboard Soyuz vehicle 28S (4/27/12) for ground-based assessment. Additional particulate and debris samples were collected from filter element inlet screens during ISS Increment 31 using the ISS portable vacuum cleaner.¹ A vacuum cleaner bag filled with debris was returned in early July 2012 aboard Soyuz vehicle 29S (7/1/12) ground-based assessment. In addition to the vacuum cleaner bag, the samples returned aboard Soyuz vehicle 29S included a swatch of FGB panel material and the vacuum cleaner high efficiency particulate air (HEPA) filter element.

Particular points of interest for assessing the samples returned aboard Soyuz vehicles 28S and 29S were for the presence of allergens, CDRA zeolite dust, and FGB panel fibers. All of the samples were delivered initially to the microbiology laboratory at the National Aeronautics and Space Administration's (NASA) Johnson Space Center (JSC) to allow a portion of the materials to be collected for allergen evaluation. Results from the allergen evaluation are reported elsewhere.² Once materials necessary for the allergen study were collected, the samples were delivered to NASA's Marshall Space Flight Center (MSFC) for physical classification, optical microscopy and photographic

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analysis, and scanning electron microscopy (SEM) evaluation using energy dispersive X-ray spectrometry (EDS) methods. The following discussion describes the results from the physical classification, optical microscopy and photographic analysis, and SEM EDS analysis.

II. Sample Characteristics

The samples returned aboard Soyuz vehicle 28S for ground-based assessment were collected using Kapton[®] tape and labeled as follows:

- 1) "Sleep Station Dust NOD 2 D_5 UT 066"
- 2) "PMA1 Cone Screen GMT 2012/110"
- 3) "Node3_D3_01 Bacteria Filter Sample 1 Square Area GMT 054/2012 (Filter was last vacuumed ~2 weeks prior on GMT 042)"

These Kapton[®] tape samples were folded upon themselves making analysis difficult. Some loose particulate matter was found in the bags containing the tape samples. These loose particles were collected for SEM EDS analysis. The sleep station dust sample bag contained two tape samples. One was removed and used for the allergen analysis and one was delivered to NASA MSFC for physical and chemical characterization. Figure 1 shows the samples returned aboard Soyuz vehicle 28S.

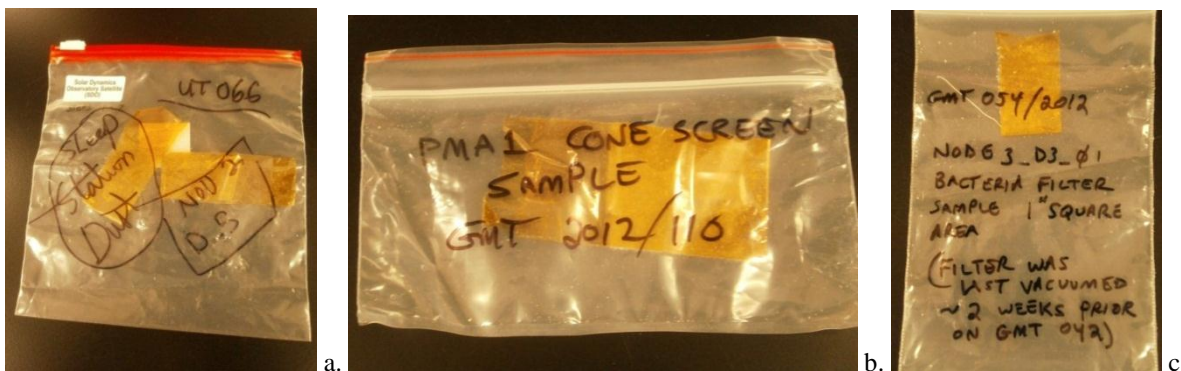


Figure 1. Kapton[®] tape samples returned aboard 28S. Depicted are tape samples collected from a.) the sleep station, b.) the PMA1 cone screen, and c.) a Node 3 bacteria filter element face.

The items returned aboard Soyuz vehicle 29S for ground-based analysis were the following:

- 1) Vacuum cleaner bag containing mixed debris.
- 2) Vacuum cleaner HEPA filter element with Kapton[®] tape over the inlet face.
- 3) FGB panel fabric swatch.

The tape over the vacuum cleaner HEPA filter inlet face removed most of the debris from the face so analysis had to be approached similarly to the Kapton[®] tape samples returned aboard Soyuz vehicle 28S. The vacuum cleaner bag as received had been cut open to collect materials for the allergen study. Blue-grey lint was the predominant material in the vacuum bag. The FGB panel fabric swatch measured approximately 3.8 cm wide \times 9.5 cm long. A portion measuring approximately 1.9 cm wide \times 3.8 cm long was removed for the allergen study before delivery to NASA MSFC. The FGB panel fabric swatch appeared to be a "dirty white" color with the appearance of dark mildew-like stains. A "yellowing" pattern within the fabric appears to indicate that water may have "wicked" into the fabric and then evaporated leaving a "water stain" pattern. Figure 2 shows the samples returned aboard Soyuz vehicle 29S.

III. Assessment Methods

The samples were evaluated as received and methods appropriate for analyzing them were selected. The samples collected on Kapton[®] tape were found to be best analyzed by the following methods:

- 1) Optical microscopy.
- 2) SEM EDS spectral scans of broad tape views with selected scans of individual materials.
- 3) SEM EDS spectral scans of individual particles collected on SEM stubs from the sample transportation bags.

The SEM EDS method helps to determine an assessment target's elemental composition by identifying specific elements comprising the target and their relative mass proportions. Comparing SEM EDS scan results to a reference material sample or a material EDS spectral database can assist in identifying unknown materials. Additional

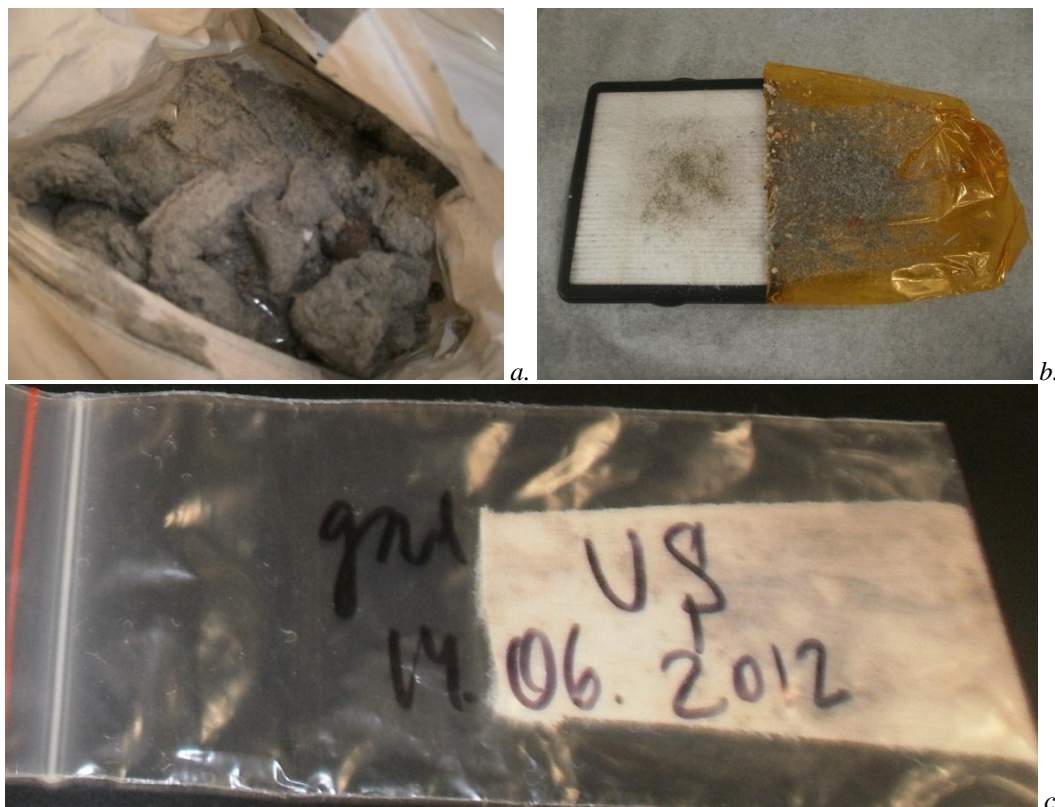


Figure 2. Debris samples and fabric material returned aboard 29S. Views of a.) the mixed debris in the vacuum cleaner bag, b.) the vacuum cleaner HEPA filter, and c.) the FGB panel material swatch.

information on physical and chemical properties of the material under evaluation must be considered to truly determine the material's identity.

Tape samples present challenges to SEM EDS analysis due to the dominant presence of the tape itself compared to the fiber and granular particulate debris covering the tape. Also, particles and rough surfaces without comparative standards available such as those that are the subject of this assessment have an accuracy of about $\pm 5\%$. All efforts were made when possible to isolate individual particulate debris items from the sample transportation bags that were similar in appearance to material subjected to focused analysis while still attached to the tape. The tape samples do afford the opportunity to search for particles that may exhibit elemental composition similar to CDRA zeolite pellet dust. CDRA zeolite pellets were analyzed independently to establish an elemental composition basis for comparison. Full tape sample SEM EDS spectral scans were conducted and the backscatter imaging elemental contrast effect was observed to screen for individual particles with high elemental proportional intensities similar to CDRA zeolite pellets. Selected particles were then probed individually. A similar approach was conducted for particles collected from the tape sample transportation bags. The methods used for evaluating the tape covering the vacuum cleaner HEPA filter returned aboard Soyuz vehicle 29S were the same as those applied to the tape samples returned aboard Soyuz vehicle 28S.

The vacuum cleaner bag debris was evaluated using the following methods:

- 1) Size classification using the following standard test sieves: No. 14 (1,400 μm), No. 18 (1,000 μm), No. 35 (500 μm), No. 60 (250 μm), No. 100 (150 μm), No. 140 (106 μm), No. 200 (75 μm), No. 270 (53 μm), and No. 500 (25 μm).
- 2) Mass fraction determination of the materials in each size class range using a laboratory digital scale and analytical balance.
- 3) Optical microscopy and photography.

The vacuum cleaner HEPA filter inlet face was evaluated using optical microscopy and photography. SEM EDS, optical microscopy, and photography were used to evaluate the FGB panel fabric swatch.

IV. Results and Discussion

The following discussion provides details of ISS debris analysis results and discusses key observations.

A. Tape Samples Returned Aboard Soyuz Vehicle 28S

Evaluation of the tape samples returned aboard Soyuz vehicle 28S focused on visual observations via optical microscopy and whether particles exhibiting chemical composition similar to CDRA zeolite pellets might be present based on SEM EDS methods. Visual observations noted a variety of granular and fibrous material on the tape. As shown by Fig. 3, the tape sample from the Node 3 bacteria filter contains various fiber colors, however, white fibers predominate. Granular material appears to be composed of human skin, nail clippings, and food debris. This sample was typical of all the tape samples. It was noted that the tape sample from the sleep station contained more human hair strands while the pressurized mating adapter-1 (PMA1) cone screen sample had more blue-grey lint than the other tape sample locations. Individual particles were collected from the tape samples and placed on SEM stubs for analysis with attention given to elemental composition that may indicate the presence of CDRA zeolite pellet material.

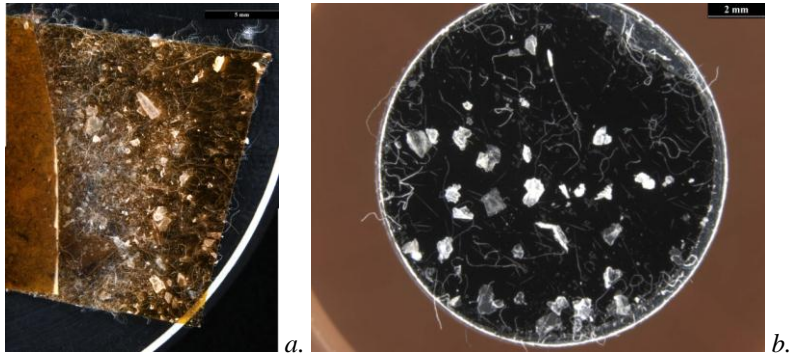


Figure 3. Node 3 bacteria filter tape sample and SEM stub containing debris. Typical for the tape samples returned aboard Soyuz vehicle 28S.

CDRA zeolite pellets were evaluated by SEM EDS using eight different views that included evaluating fractured pellet surfaces and un-fractured pellet surfaces. This allowed for a range of possible composition through a particle's cross section to be established as a basis for comparison to the tape sample SEM EDS and backscatter imaging elemental contrast observation results. The elemental composition of CDRA zeolite pellets was found to primarily consist of silicon (16 wt%-19 wt%), aluminum (16 wt%-19 wt%), oxygen (37 wt%-41 wt%), calcium (8 wt%-14 wt%), magnesium (1.2 wt%-2.6 wt%), sodium (1 wt%-2 wt%), and carbon (9 wt%-13 wt%). Lesser quantities of phosphorus, sulfur, potassium, iron, and titanium were observed.

Based on the backscatter imaging elemental contrast observations, ninety individual particles were selected and probed using SEM EDS. The results indicated elemental compositions high in carbon, oxygen, and silicon. Neither the backscatter imaging elemental contrast observations nor the individually probed particle results isolated material possessing a composition consistent with CDRA zeolite pellet material. Most particles indicate a closer resemblance to organosilicone compounds due to their high carbon, oxygen, and silicon content.

B. Vacuum Cleaner HEPA Filter Tape Debris

The Kapton[®] tape covering the vacuum cleaner HEPA filter pulled the accumulated debris off the filter. Optical microscopy found debris adhering to the tape to consist of skin fragments, nail clippings, food crumb debris, and a variety of hair and fibrous debris. These materials were similar to those observed on the tape samples returned aboard Soyuz vehicle 28S. Two sections were cut from the tape for SEM EDS analysis. Scans and targeted EDS



Figure 4. Cut sections of Kapton[®] tape covering the vacuum HEPA filter element. Significant quantities of skin fragments, nail clippings, granular debris, hair, and fibrous debris are evident.

analysis were conducted on both sections of tape. Seventeen individual particles were selected for targeted SEM EDS analysis. Figure 4 shows the tape cutout samples mounted for SEM EDS analysis. Skin fragments, nail clippings, granular debris, and a variety of hair and fibrous debris are

visible. The EDS analysis of seventeen targeted particles shown by Fig. 5 found the elemental composition to be dominated by carbon and oxygen. Some observation to note were particle number seven which contained zinc (31 wt%), titanium (6.7 wt%), aluminum (2.4 wt%), magnesium (1.5 wt%), silicon (2 wt%), phosphorus (12.3 wt%), oxygen (28.7 wt%), carbon (14.5 wt%), and lesser amounts of iron, nickel, potassium, sulfur, and chlorine. Particle number eleven was reported to contain aluminum (86 wt%), magnesium (4.8 wt%), and zinc (3.7 wt%) with lesser quantities of nickel, silicon, chromium, iron, copper, oxygen, and carbon. Analysis of particle number nine found high quantities of iron, carbon, and oxygen. Comparison of all SEM EDS results found no spectral signatures consistent with CDRA zeolite pellet material.

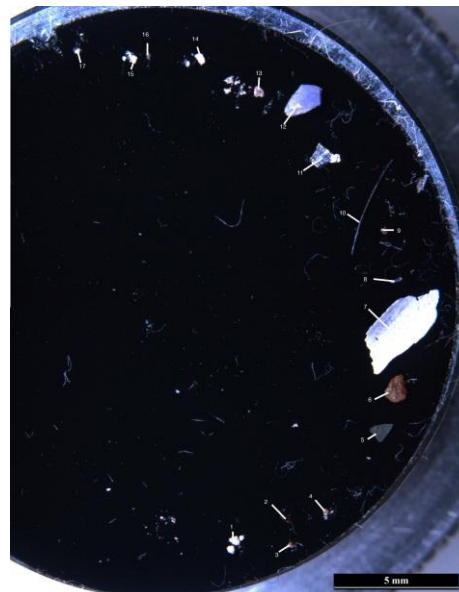


Figure 5. Selected particles from the vacuum cleaner HEPA filter. Individual particle composition was found to be primarily carbon and oxygen.

C. Vacuum Cleaner Bag Debris

1. Vacuum Bag Debris Separation and Classification

The vacuum cleaner bag debris was assessed using separation, optical microscopy, and photography methods. The bag, as received, had been cut open and emptied to allow for samples to be collected for allergen assays. The debris was replaced into the bag and the bag was taped closed. The bulk material is best described as blue-grey fibrous debris with human hair, food, paper, plastic, and miscellaneous granular debris mixed within it. The vacuum bag containing the debris weighed 169.2 grams as received. The empty vacuum cleaner bag weighed 93.2 grams. Based on the bag's weight difference when full and empty, the total debris weight was approximately 76 grams. The total debris was transferred directly into a pre-weighed plastic beaker (58.9 grams). The total weight of the beaker and debris was 134.8 grams. Subtracting the beaker weight, the debris weight was 75.9 grams which agrees with the debris weight derived from the vacuum cleaner bag weight measurements. After establishing the total debris weight, standard test sieves and forceps were used to separate the debris into different size and type fractions. Each fraction was weighed. Table 1 summarizes the observed weights for each size and type fraction to <500 μm .

All of the debris was transferred to a No. 14 test sieve which has 1,400 μm apertures. The material was agitated gently by hand. The material retained on the No. 14 test sieve and the material that passed through the sieve weighed 67.76 grams and 8.19 grams, respectively. There was 99.9% mass accountability for this sieving operation.

The material retained on the No. 14 test sieve consisted of large blue-grey lint balls with hair and long fibers interspersed within it. Food, paper, and plastic debris were also observed. Unusual items found in this debris included a small plant seedling, a slender wooden dowel that may be a toothpick fragment, a small screw that possibly came from eyeglasses, and a thin wire spring. These items are shown by Fig. 6. The material retained on the No. 14 test sieve was classified into four fractions shown by Fig. 7 — lint and hair (38.55 grams), paper and

Table 1. Vacuum cleaner bag debris classification by mass. Debris type and mass fraction after size classification using No. 14, No. 18, and No. 35 standard test sieves.

SIZE RANGE (μm)	CLASSIFICATION	WEIGHT (grams)	FRACTION (wt%)
>1,400	Lint and hair	38.55	50.9
	Paper and plastic	3.86	5.1
	Food debris	24.35	32.2
	Residue	1.00	1.3
>1,000 and <1,400	Granular debris	0.98	1.3
	Lint and hair	0.65	0.9
>500 and <1,000	Granular debris	1.59	2.1
	Lint and hair	0.71	0.9
<500	Mixed granular/fibrous	4.00	5.3

plastic (3.86 grams), food debris 24.35 grams), and granular residue (1.00 grams). This additional classification maintained nearly 99.9% mass accountability.

The material (8.19 grams) that passed through the No. 14 test sieve consisted of granular material, smaller fibrous debris, and smaller hair fibers. This material was transferred to a No. 18 test sieve which has 1,000 μm apertures. The sieve was agitated gently by hand. The material retained on the No. 14 sieve and the material that passed through the sieve weighed 1.63 grams and 6.33 grams, respectively. There was 97.2% mass accountability for this sieving operation. The material that passed through the No. 18 test sieve was similar in appearance to the material retained on the sieve. Both fractions consisted of granular material, fibrous debris, and hair fibers but of different sizes. The granular and fibrous debris fractions for the material retained on the No. 18 test sieve, shown by Fig. 8, weighed 0.98 grams and 0.65 grams, respectively.

The material (6.33 grams) that passed through the No. 14 test sieve was transferred to a No. 35 test sieve which has 500 μm apertures. The sieve was agitated gently by hand. The material retained on the No. 35 test sieve weighed 2.30 grams while the material that passed through the sieve weighed 4.00 grams. There was 99.5% mass accountability for this sieving operation. The granular and fibrous fractions for the material retained on the No. 35 test sieve, shown by Fig. 9, weighed 1.59 grams and 0.71 grams, respectively. The material that passed through the No. 35 test sieve, shown by Fig. 10, consisted of a fine granular material with small fibrous debris and hair dispersed within it.

The material that passed through the No. 35 test sieve was subjected to further size classification using a test sieve series with mechanical agitation. The procedure was adapted from Ref. 3. The sieve series consisted of a No. 60 test sieve (250 μm aperture), No. 100 test sieve (150 μm aperture), No. 140 test sieve (106 μm aperture), No. 200 test sieve (75 μm aperture), No. 270 test sieve (53 μm aperture), and No. 500 test sieve (25 μm aperture).

The test sieve stack was mechanically agitated for 5 minutes and then the collection pan was checked. There was no material found in the collection pan. The stack was agitated for another minute with no material collected in the pan. Inspection of each test sieve found that measurable quantities of the material transferred as far as the No. 200 test sieve (75 μm aperture). A residual quantity of material was observed in the No. 270 test sieve (53 μm aperture). No material was observed below the No. 270 test sieve. The top test sieve (No. 60, 250 μm aperture) was weighed and then several 1-minute agitation periods were conducted. The No. 60 test sieve was weighed between each 1-minute agitation segments until <1% change in the No. 60 test sieve weight was demonstrated for three successive agitation periods. The final percent difference was found to be approximately 0.07% so the sieve analysis operation was determined to be complete. The cumulative agitation time was 10 minutes.

Table 2 summarizes the size classification results for the debris <500 μm in size. The total material weight after removing it from a storage bag and completing the sieving operation was 3.984 grams compared to the earlier 4.00 grams that was retained on the No. 35 test sieve. The mass accountability was 99.6% for this sieving operation.

Table 2. Size classification of debris <500 μm . Debris type and mass fractions after size classification using a standard test sieve series with mechanical agitation.

SIZE RANGE (μm)	CLASSIFICATION	WEIGHT (grams)	FRACTION (wt%)
>250 and <500	Mixed granular/fibrous	1.353	33.96
>150 and <250	Mixed granular/fibrous	1.763	44.25
>106 and <150	Mixed granular/fibrous	0.801	20.10
>75 and <106	Mixed granular/fibrous	0.066	1.66
>53 and <75	Granular residue	0.001	0.03
>25 and <53	None observed	0	0
<25	None observed	0	0

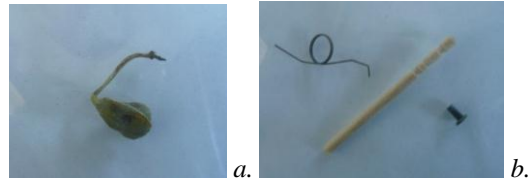


Figure 6. Interesting items retrieved from the vacuum cleaner bag debris. Items that were different from the typical debris included a.) a plant seedling, and b.) a toothpick fragment, a small screw, and a wire spring.

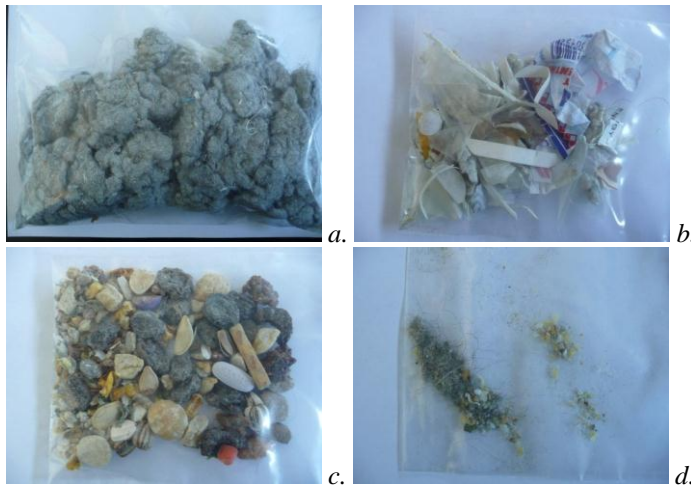


Figure 7. Debris retained on the No. 14 standard test sieve. Debris classifications include a.) lint and hair, b.) paper and plastic, c.) food debris, and d.) granular and fibrous residue.

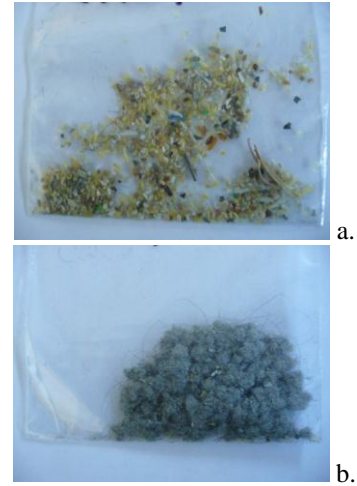


Figure 8. Debris retained on the No. 18 standard test sieve. Material fractions include a.) granular debris and b.) fiber and hair debris.

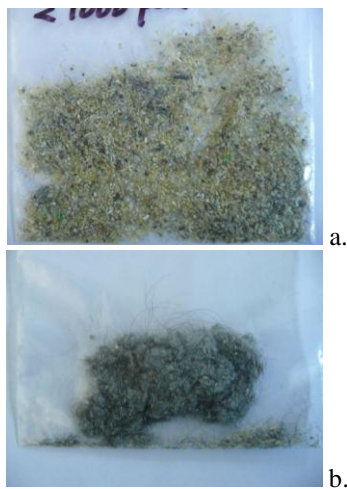


Figure 9. Debris retained on the No. 35 standard test sieve. Material fractions include a.) granular debris and b.) fiber and hair debris.



Figure 10. Debris that passed through the No. 35 standard test sieve. Mixed granular, fiber, and hair debris.

2. Vacuum Cleaner Bag Debris Appearance and Observations

The material retained on the No. 14 sieve ($>1,400\ \mu\text{m}$) shown by Fig. 7 consisted of four classifications—fiber and hair, food, paper and plastic, and residue. The fiber debris was blue-grey lint with human hair dispersed throughout it. The lint retained particles of various sizes which can be dislodged by handling the bulk material. The appearance demonstrates that lint accumulation can, in itself, serve as an effective particulate filter. The food debris consisted of dried raisins, nut shells (e.g. pistachio and sunflower), nut fragments (e.g. peanut, macadamia, sunflower), dried vegetables (e.g. carrot, corn, pea, and potato), leaf fragments, a multivitamin tablet, a pretzel or potato stick fragment, large cookie or brownie fragments, a candy-coated chocolate fragment, and a large array of granular crumbs. The granular crumbs are difficult to ascertain their origin and a detailed forensic analysis may be beneficial. The paper and plastic debris consisted of bandage wrapper fragments, labels, personal care product wrapper fragments, plastic packaging fragments, elastic seal material fragments, and a variety of other debris. The residue consisted of material similar to the granular food crumbs mixed with fibers and hair.



Figure 11. Granular debris retained on the No. 18 test sieve. a.) Typical debris showing dried skin and other unidentified debris at 10X magnification b.) a view showing a fruit stem and a large nail clipping at 8X magnification c.) a view showing a mechanical pencil lead fragment at 10X magnification.

The material retained on the No. 18 sieve ($>1,000 \mu\text{m}$ and $<1,400 \mu\text{m}$) shown by Fig. 8 consisted of two classifications—granular and fibrous debris. The granular debris consisted primarily of large dried skin fragments and nail clippings. A stem, possibly from a cherry, was found. A mechanical pencil lead fragment was also found. Figure 11 shows some typical granular debris in this size fraction. Small plastic fragments were observed. The fibrous debris consisted of smaller blue-grey lint fibers and human hair.

The material retained on the No. 35 test sieve ($>500 \mu\text{m}$ and $<1,000 \mu\text{m}$) shown by Fig. 9 consisted of two classifications—granular and fibrous debris. The granular debris consisted of smaller dried skin fragments and nail clippings. Small mechanical pencil lead fragments were also found. Figure 12 shows detailed views of the granular material retained on the No. 35 test sieve. The fibrous debris consisted of fine blue-grey lint and short human hair fragments.

The material that passed through the No. 35 test sieve ($<500 \mu\text{m}$) shown by Fig. 10 consisted of a uniform tan-colored, powdery mixture of granular material and short human hair fragments. The granular material was similar in color to the dried skin fragments and nail clipping found in the larger sized granular material fractions; therefore, it may be concluded that this fine granular material may be fine skin and nail clipping fragments. Figure 13 shows a more detailed view of the material that passed through the No. 35 test sieve.

The material that passed through the No. 35 test sieve was further evaluated for its size fraction by passing the material through a test sieve series. The material retained on the No 60 test sieve ($>250 \mu\text{m}$ and $<500 \mu\text{m}$) consisted of granular material with human hair and fine fibers dispersed within it. Blue-grey lint was observed in this size fraction. The material retained on the No. 100 test sieve ($>150 \mu\text{m}$ and $<250 \mu\text{m}$) consisted of finer granular material with human hair dispersed within it. No blue-grey lint was observed in this fraction. The fraction retained on the No. 140 test sieve ($>106 \mu\text{m}$ and $<150 \mu\text{m}$) consisted of granular material with a few human hair fibers dispersed within it. Likewise, the fraction retained on the No. 200 test sieve ($>75 \mu\text{m}$ and $<106 \mu\text{m}$) consisted of granular material

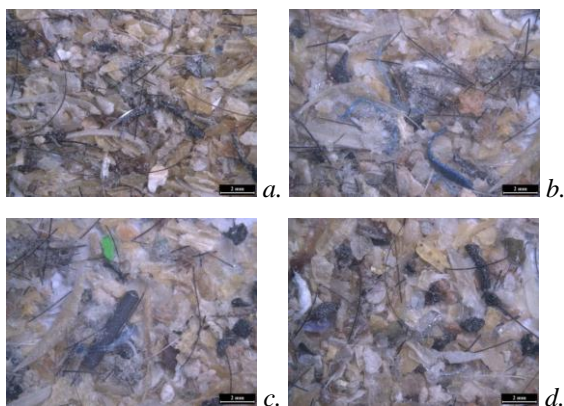


Figure 12. Granular debris retained on the No. 35 test sieve. Views at 10X magnification showing a.) debris with dried skin and nail clippings, b.) blue fibers possibly from Velcro® closures, c.) mechanical pencil lead, blue fibers, and nail clippings, and d.) metal turnings.

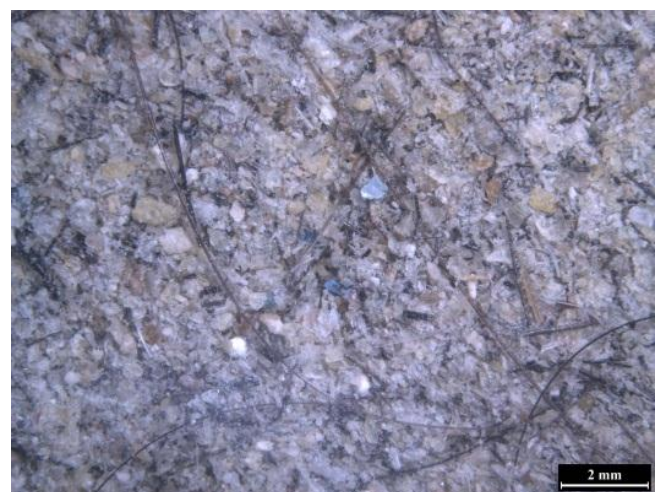


Figure 13. Debris that passed through the No. 35 test sieve. The debris at 10X magnification shows mixed granular material and human hair.

with a few human hair fibers. The residue isolated on the No. 270 test sieve ($> 53 \mu\text{m}$ and $<75 \mu\text{m}$) consisted of granular material.

3. Vacuum Cleaner HEPA Filter Inlet Face Analysis

The vacuum cleaner filter was assessed using optical microscopy and photography. Figure 14 shows a view of the filter inlet face. The white to clear fibers comprising the vacuum cleaner filter's pre-filter media are interesting because they appear very similar to white fibers observed in the vacuum cleaner bag debris.

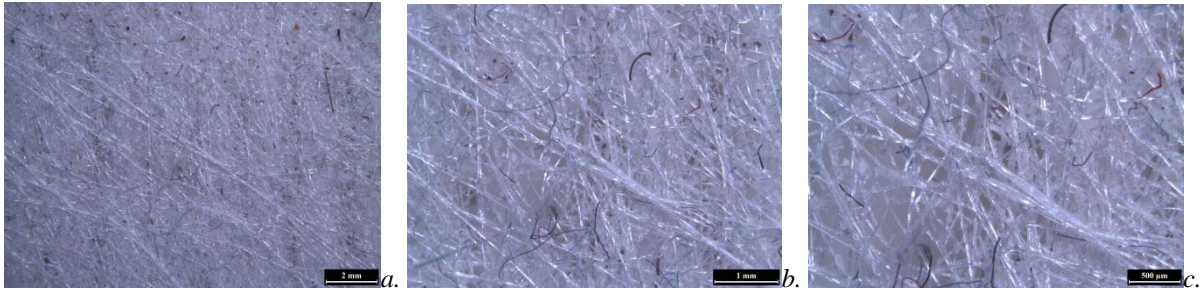


Figure 14. Vacuum cleaner bag filter optical microscopy photographs. Views at a.) 10X, b.) 25X, and c.) 40X magnification show white to clear fibrous material composition with some captured granular and fibrous debris. The Kapton[®] tape covering the filter face removed the major portion of accumulated debris from the filter face.

4. FGB Panel Fabric Swatch Evaluation

The FGB panel fabric swatch was evaluated by SEM EDS, optical microscopy, and photography. Seventeen areas shown by Fig. 15 were probed using SEM EDS. Both fibers and debris adhering to the fibers were targeted. The fabric consisted of three types of fibers. Each fiber was targeted as well as fourteen areas where debris adhered to the fabric. The targeted fibers were found to have an elemental composition to consist primarily of fluorine (32.8 wt% average), carbon (51.5 wt% average), oxygen (11.3 wt% average), and nitrogen (4.7 wt% average). Lesser quantities (0.2 wt% to 1 wt% range) of calcium, sodium, magnesium, aluminum, silicon, chlorine, potassium, and titanium were observed. These observations may be indicative of a fluoropolymer and acrylic fiber materials. The elemental composition of the debris was found to consist of fluorine (22 wt% average), carbon (44.8 wt% average), oxygen (15.8 wt% average), and nitrogen (5.9 wt% average). Lesser quantities (0.5 wt% to 2.6 wt%) of calcium, sodium, magnesium, aluminum, silicon, phosphorus, sulfur, chlorine, potassium, iron, and titanium were observed. The particle probed at location number 8 contained 4.1 wt% zirconium. Similarities are noted in the primary elements observed for all seventeen probed locations. This is likely due to the dominant fiber background of the debris adhering to the fibers.

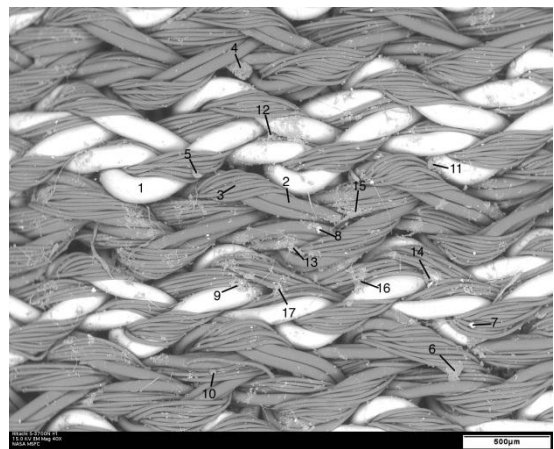


Figure 15. FGB panel fabric swatch SEM image showing seventeen EDS probed locations. Three fiber types are evident at 40X magnification of the smooth side of the FGB panel fabric. Fibrous and particulate debris adhering to the surface are visible.

Both sides of the fabric were evaluated using optical microscopy. Figure 16 shows views of the fabric's smooth side and backing side. The three fiber types are visible on the fabric's smooth side. These fibers consist of a clear fiber, a multi-strand twisted fiber, and a thin white fiber. The backing side consists of a single type of fiber that appears to be the thin white fiber visible in the smooth fabric side weave. Physical inspection found the fabric to be flexible with no indication of brittleness and no tendency to produce dust. A few white backing fibers were found loose in the sample bag but the number of loose fibers was <50 and may have been shed from the cut edges. No dust or other debris was found in the sample bag. The fabric was inspected by hand and abraded by vigorously rubbing the back with the fingers. Less than 20 fibers were shed from the material after a minute. Based on these observations it may be concluded that fibers originating from the FGB panel fabric material would be released only when the material is cut or subjected to repeated mechanical abrasion rather than spontaneously generated.

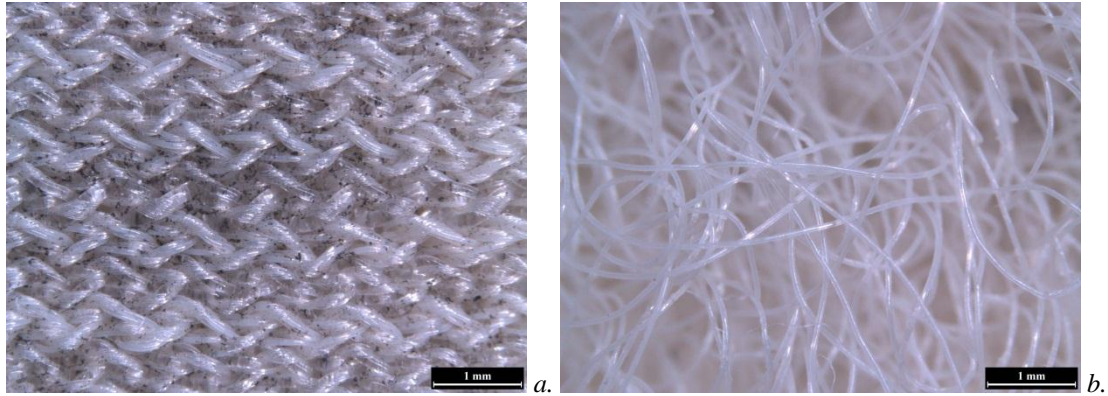


Figure 16. FGB panel fabric swatch optical microscopy images. Views at 25X magnification showing a.) three fiber types consisting of a clear fiber, thin white fiber, and a multi-strand twisted fiber are visible on the fabric smooth side. Dark spots in the image are the mildew-like stain on the fabric, b.) white fiber loops are visible on the fabric back. These fibers are visible in the weave on the fabric's smooth side.

5. Fibrous Debris Observations

Fibrous debris was found to be predominantly clothing lint and human hair. Other fibrous debris was observed as shown by Fig. 12. Scanning the granular debris from all the size fractions indicated some white fibers of interest shown by Fig. 17. These fibers strongly resembled the multi-strand fiber found in the FGB panel fabric (Fig. 17, view a.) as well as the white fiber loops on the back of the FGB panel fabric and the white fibers on the vacuum cleaner HEPA filter face. The fibers on the back of the FGB panel fabric and the fibers on the vacuum cleaner HEPA filter face are very similar in appearance as shown by Figs. 14b and 16b. While Fig. 17 shows that there are fibers in the vacuum cleaner bag debris which are similar in appearance to FGB panel materials, the similarity between the FGB panel and vacuum cleaner HEPA filter face fibers confounds a source determination based strictly on appearance. More detailed chemical and physical analysis by forensic science fiber analysis experts.



Figure 17. Vacuum cleaner bag debris showing fiber debris of interest. Views at 10X magnification showing a.) a multi-strand fiber having similarity to an FGB panel fabric multi-strand fiber, b.) white fibers similar to those observed on the FGB panel fabric back or the vacuum cleaner bag HEPA filter face, and c.) more white fibers similar to those observed on the FGB panel fabric back or vacuum cleaner bag HEPA filter face.

V. Comparison to Previous Crewed Spacecraft Debris Evaluations

Previous efforts to characterize the suspended particulate load aboard crewed spacecraft were conducted during the Space Shuttle Program both during flight and post-flight. Despite differences in the sampling methods employed, comparison of the ISS debris composition with results from past characterization work shows marked similarities described by the following discussion. These similarities are thought to exist because the pre-flight materials selection and control methods as well as the type of fabrics, food, equipment, and activities that may generate particulate debris aboard the ISS have significant heritage carryover from the Space Shuttle Program.

A particulate sampler was used to collect particles in several size ranges— $<2.5 \mu\text{m}$, $2.5\text{-}10 \mu\text{m}$, $10\text{-}100 \mu\text{m}$, and $>100 \mu\text{m}$ during mission STS-32.⁴ Chemical composition was also assessed for the particulate matter collected. Of the particulate material collected, the size fraction in the $10\text{-}100 \mu\text{m}$ accounted for 9.75% of the total reported average mass concentration. This is comparable to the 8.36 wt% found in the ISS debris fraction $<150 \mu\text{m}$.

Chemical composition of the particles collected during mission STS-32 indicated carbon, silicon, aluminum, sodium, chlorine, and potassium as the predominant elements with carbon and silicon being the most prevalent. These results, again, are similar to those observed during the ISS debris assessment that found carbon- and silicon-rich materials.

Debris assessment was conducted post-flight for mission STS-13.⁵ This assessment included an analysis of debris collected using the Shuttle vacuum cleaner. The vacuum cleaner debris analysis found 51.9 wt% fibers, 32.2 wt% miscellaneous debris including food, and 15.9 wt% paper and plastic debris. This result is very comparable to the ISS debris assessment findings summarized by Table 1.

A post-flight forensic analysis of debris collected from filters the Spacelab Life Sciences-1 (SLS-1) module was conducted in 1991.⁶ Similar to the vacuum cleaner bag debris from STS-13 and the ISS, this assessment found the debris consisted of 50-55% fibers. This range encompasses the observed fiber content of debris examined from STS-13, 51.9 wt%, and the ISS, 52.7 wt%.

VI. Further Evaluation

The vacuum cleaner bag, HEPA filter, and the granular debris fraction <106 μm are subjects for additional evaluation by NASA's Jet Propulsion Laboratory (JPL) and Ohio State University. Specific details regarding these additional evaluations are reported elsewhere.

Researchers at JPL developed a method to better evaluate viable microorganism frequency, diversity, and distribution within particulate and fiber debris returned from the ISS. The vacuum cleaner bag and HEPA filter were subjected to NASA JPL's method that uses a propidium monoazide (PMA) treatment followed by downstream quantitative polymerase chain reaction (qPCR) and pyrosequencing analysis.⁷ The analysis provided insight with respect to background microbiological contamination beyond that attained using traditional microbiological assay methods.

Researchers at Ohio State University (OSU) were provided debris samples for the size range <106 μm . This debris fraction was analyzed to determine its full composition using Fourier transform infrared (FTIR) spectroscopy. The method involves dispersing the particulates onto a mesh and taking 512 scans per pixel to map the area. Then the peaks in the spectra are used to obtain information on the chemical bonds in the dust material to assist in determining composition.

Additional work that may be useful to future spacecraft cabin particulate and debris control includes developing common particulate and debris sampling methods as well as conducting debris particle counts for each size distribution. Conducting particle counts to determine how they compare with both size and mass distribution can provide improved insight for developing active particulate and debris control functional specifications and requirements for future exploration class vehicles.

VII. Summary

Debris samples that were collected for ground-based analysis using Kapton[®] tape were returned aboard Soyuz vehicle 28S. At a later date a vacuum cleaner bag containing mixed debris, a vacuum cleaner HEPA filter element, and a swatch of FGB panel fabric were returned aboard Soyuz vehicle 29S. These samples and materials were assessed using SEM EDS, optical microscopy, photography, and physical separation techniques. The debris materials were found to consist of four primary classes—lint and hair, paper and plastic debris, food debris, and granular debris. The debris was analyzed to search for possible dust from the CDRA zeolite material and fibrous debris that may originate from FGB panel fabric. Findings indicated no definitive evidence of CDRA zeolite material or fibrous debris from FGB panel fabric. The debris' element composition was found to consist predominantly of carbon, oxygen, and silicon. Fiber debris was the most prevalent at 52.7 wt% followed by food debris at 32.2 wt%, paper and plastic debris at 5.1 wt%, and granular debris at 10 wt%. Comparison to assessments conducted during the Space Shuttle program indicates consistency in debris composition.

VIII. Conclusion

Analysis of the samples and debris returned aboard Soyuz vehicles 28S and 29S found that debris that may be controlled by the crewmembers such as clothing lint; hair and nail clippings; and food, paper, and plastic debris accounted for 90 wt% of the total debris contained in the vacuum cleaner bag. Fabric lint and fibrous debris accounted for nearly 54% of the total debris weight. Food, paper, and plastic debris accounted for 37.3 wt%. Skin fragments, nail clippings, and other granular debris accounted for 8.7 wt% of the total debris. Size classification did not isolate material <53 μm in size. The smallest size fraction retained on the No. 270 test sieve (53 μm aperture) weighed approximately 1 milligram or 0.0014 wt% of the total vacuum bag material weight. Comparison to

previous crewed spacecraft debris assessments found striking similarity, particularly with regard to the weight percentage contributed by fibrous debris.

The vacuum cleaner bag and tape sample analysis observed no evidence of particulate debris consistent with CDRA zeolite pellets. Fibrous debris similar to fibers found on the FGB panel back and the vacuum cleaner HEPA filter face were observed in the debris <1,400 µm in size. However, similarity between the fibers found on the FGB panel back and the vacuum cleaner HEPA filter confounds identification by observation. More extensive analysis by forensic fiber analysis experts would be necessary to identify the fiber materials. This fibrous debris was insignificant in quantity compared to all other debris.

The debris evaluation leads to considerations for future ISS operations and future crewed spacecraft design and flight operations. Conducting frequent, thorough housekeeping operations aboard crewed spacecraft is necessary to address cumulative lint and debris buildup. Consideration should be given to selecting clothing and towel fabrics that produce very little lint. Similarly, consideration should be given to selecting foods that produce less packaging and crumb debris.

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